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The Groningen AMS tandetron

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Abstract

The Groningen tandetron-AMS is a fully automated ^{14}C -mass spectrometer with a maximum capacity of 3000 measurements annually. In the first two years of operation we performed 4000 measurements. We routinely perform high precision measurements ($< 5\%$ for ^{14}C , and 2% for ^{13}C). Here we report on the status and performance of the Groningen machine, improvements made and planned in the near future, and highlight some research projects.

1. Introduction

The latest generation tandetron-based AMS is characterised by simultaneous injection of the three carbon isotopes [1,2]. Measuring individual isotopes under the same conditions, minimizes fractionation effects, and provides an on-line diagnostic tool for machine performance during measurements [3]. This high throughput machine is capable of dating up to 3000 samples per year. There are now 3 machines of this type in operation: Woods Hole since 1990 [4], Groningen since 1994 and very recently Kiel [5]. Since Groningen, these machines are manufactured by High Voltage Engineering Europa (HVEE) in Amersfoort, the Netherlands [6].

The system can be divided into five main sections: (1) the ion source; (2) the recombinator; (3) the tandetron tandem accelerator; (4) the high-energy mass spectrometer; and (5) the computer control system with data acquisition. Fig. 1 shows the lay-out of the machine. We repeat here a summarized description of the system. A full description of the HVEE model 4130 has been given before [1,2,7]. The high current caesium sputter-type ion source produces a 36 keV negative ion beam from graphite, which is produced from the samples [8]. Up to 59 samples can be loaded in the computer-controlled carousel. The ion beam extracted from the source (20–50 μA) is focused with an einzel lens into the recombinator. This recombinator [9] consists of four magnets. The beams with mass 12, 13 and 14 are separated here by about 2 cm. A chopper wheel reduces the ^{12}C beam by a factor of about 90 to reduce the beam load in the accelerator.

The tandem accelerator operates at 2.5 MV. At the terminal, circulating Ar-gas strips the negative carbon ions

to C^{3+} . Further analysis is done with a high-energy mass spectrometer, consisting of a 110° magnet, a 33° electrostatic deflector and a 90° magnet. After the first magnet, the ^{12}C and ^{13}C beams are measured in individual faraday cups. Typical currents for these beams are 200–500 nA. The ^{13}C cup is equipped with an integrated slit system. This setup provides a slit error signal, which is related to the beam displacement in the cup. The error signal is fed back to the terminal voltage driver to ensure high stability [6]. Finally, the ^{14}C particles are detected in an ionization chamber, which measures both energy loss (ΔE) and total energy (E).

The complete AMS system is controlled by fibre optic links to a Hewlett Packard I/O system. High-level actions can be performed by two personal computers, one for the source control and one for overall system control and data acquisition. This control system is designed for unattended automatic operation around the clock.

2. Operation and experience

The Groningen AMS was accepted in May 1994, just in time to report the first results at the 15th International Radiocarbon Conference in Glasgow, UK [10]. Here we report on operational experience, improvements and machine performance during the first two years since acceptance of the machine.

In the beginning of our operations, we used the AMS for measurements mainly during day time. It turned out that we needed more than one year to reach the state of unattended, around the clock operation. The reliability of some critical components had to be improved first. First, we had to improve the ion source, in particular components connected with the target change mechanism. The vacuum bellow construction has been replaced by a sliding seal which thus far, after thousands of target changes, has

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not shown a single failure. In order for this concept to work, ("differential pumping"), a small pump, selected for low power consumption had to be mounted in the source; it operates at 36 kV bias, supplied by the source isolation transformer. In addition, the target-holder construction in the source itself had to be improved mechanically for better cooling of the target. The front surface of the target electrode, which can be damaged after a long sputtering time, is modified with a replaceable insert. This greatly enhances flexibility during source cleaning operations. Also the original isolators were replaced by isolators made of polyethylene. The second major improvement was the vacuum feedthrough of the chopper wheel, which we replaced by a ferro-fluid rotary motion feedthrough. This improved vacuum conditions in the recombinator considerably. The chopper wheel itself has been replaced also by a better design: the cuts in the wheel are now tapered instead of straight. Third, the 3 MV driver electronics needed major revision and improvements, and also the power supply of the stripper gas turbo-pump inside the tank needed revision.

Only after the above improvements were made, unattended and around-the-clock operation became possible since the beginning of 1996. The machine is inspected off-line by a videocamera, connected to the laboratory's computer network; status of the machine and progress of measurements can be inspected from computers at other locations, such as the home of the authors.

A typical batch measurement consists of 52 targets, of which 5 backgrounds, 13 standards, and 34 unknown samples. Such a batch takes about 40 h of measuring time. The target carousel can hold a maximum of 59 samples. A few positions in the carousel are reserved for tuning – and testing – samples. The working standard for the Groningen AMS laboratory is sucrose (ANU or IAEA-C6) with reported consensus values for ^{14}C as 150.61 pMC,

and for ^{13}C as $\delta = -10.80\text{‰}$ [11]. The background targets are usually anthracite. Both sucrose and anthracite targets can be made either by the AMS preparation lines, or from CO_2 gas produced by the conventional laboratory.

In the ion source, a computer-controlled X–Y table sample movement is implemented. We use 8 different sputter positions. Per position, data acquisition is performed in time blocks of approximately 30 s. After every block, measured data are stored and the X–Y table moves the sample to the next point on the graphite surface to be sputtered. This way "cratering" is minimized. An additional advantage is that the target material can be sputtered more effectively, that is up to the point where almost no material is left. This procedure is followed for both our standard targets, which have a graphite surface of 2 mm diameter [8], as well as small samples which are pressed in target holders with 1 mm diameter.

For measurement of long batches (taking about 40 h), it is essential for the total AMS system to be stabilized for a long time (terminal voltage: 48 h, magnets: 24 h, source: 3 h) to prevent drifts. Before every batch, the system is tuned with a C6 sample. Tuning is straightforward with switchable Faraday cups in the source, recombinator (separate 12/13/14 stoppers), tandem entrance, and the cups in the high energy mass spectrometer. It is also possible to tune the ^{13}C beam over the ^{14}C path. During measurements of the batch, data quality is monitored by inspection of the spectrum from the ion detector; the target current monitored per block is a good practical measure for the quality of the graphite.

After the batch is measured, the data which are stored per block on the data-acquisition PC, are transferred to an optical disc for off-line analysis. A typical batch consists of 25 MB of data. The data analysis is performed off-line on a separate computer system. For this purpose we have written a program in Pascal. It calculates the ^{14}C ages and

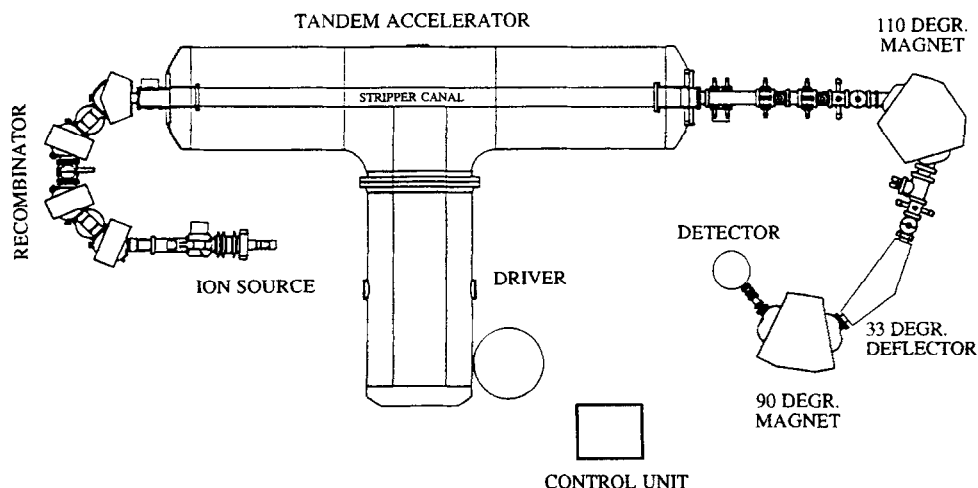


Fig. 1. Schematic lay-out of the Groningen ^{14}C -AMS system.

$^{13}\delta$ values for the samples in the batch, based on the average values for the standards and background for this particular batch. It includes full statistical treatment of the data (rejection criteria, error calculations, etc.). The AMS $^{13}\delta$ values are used for fractionation corrections. In most cases, also the $^{13}\delta$ values as measured by stable isotope mass spectrometry is known (since most organic samples are combusted in a CN-analyser, on-line with MS).

3. Performance

The first results of the Groningen AMS system were presented at the 15th International Radiocarbon Conference in Glasgow, UK. The factory acceptance test was performed on 11 sucrose targets [7]. Measurements thereafter were mainly dedicated to interlaboratory tests between the Groningen AMS and conventional laboratories, and measurements of other standards than sucrose [10]. The results from these tests yield precisions of better than 5‰ for both $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$.

Since then, we have performed a few thousand measurements more and reached a situation of operation around-the-clock. Results for the C6 standards of a typical batch measurements are shown in Fig. 2. In the top of Fig. 2, the results for ^{14}C are shown as fraction modern (pMC/100); in the bottom of Fig. 2, the results (for the same targets) for ^{13}C are shown, calculated as $^{13}\delta$. These AMS- $^{13}\delta$ values are included in the ^{14}C results as fractionation correction. The results for this batch are: 150.60 ± 0.35 pMC for ^{14}C (top of Fig. 2), and $-10.4 \pm 1.1\%$ for ^{13}C (bottom of Fig. 2). The error bars indicated correspond to the counting statistics. We note here that for small samples (using 1 mm diameter targets) the precision is usually worse.

In Fig. 3 we show the complete history of C6 (sucrose) standard targets measured over the last two years since acceptance. Close to 900 measurements are shown in this plot. The top trace (circles) shows the $^{14}\text{C}/\text{C}$ ratio and can be considered indicative for machine performance. The bottom trace (squares) is the ^{14}C fraction modern (pMC/100), corrected for isotopic fractionation by $^{13}\delta$. Deviation from the average value is generally understood by either machine or target-related problems. For this grand total, the standard deviation for these fraction modern numbers is about 5‰.

In Fig. 4, we show a comparison between regular (2 mm) and small size (1 mm) targets, measured in one batch. All targets were IAEA-C6 standards; the first half of the batch consisted of 1 mm targets, the second half of 2 mm targets. The top part of Fig. 4 shows the ^{14}C measurements (plotted in fraction modern) and the bottom part the $^{13}\delta$ value for the same targets. For ^{14}C , the standard deviation is 0.0035 (2 mm) respectively 0.0065 (1 mm); for ^{13}C , the standard deviation is 1.2‰ in $^{13}\delta$.

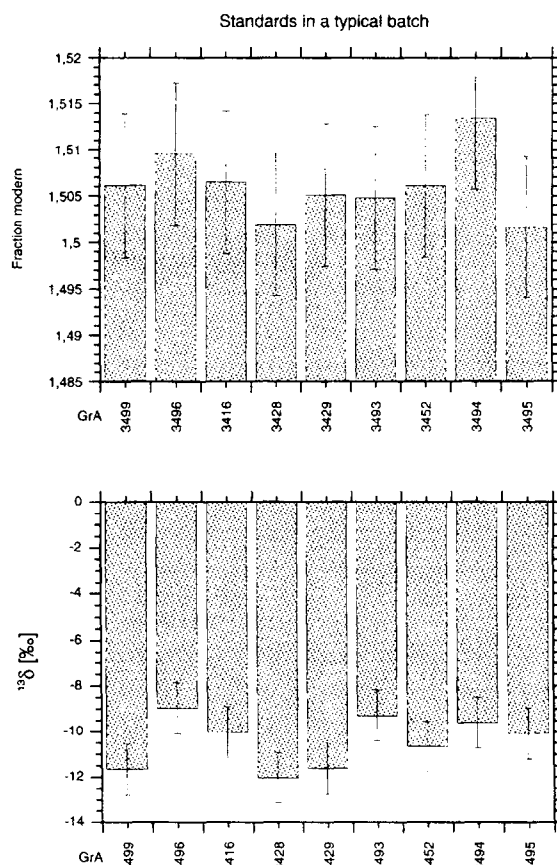


Fig. 2. ^{14}C and ^{13}C results for C6 (sucrose) standards in typical batch measurement.

Finally, we have shown that the background targets have a $^{14}\text{C}/\text{C}$ ratio of around 10^{-16} or 45 ka [8]. Target preparation is clearly the limiting factor. A “machine blank” (carbon-free target) yields a background corresponding to 100 ka [10].

4. Research

The Groningen AMS is dedicated to ^{14}C research. About 25% of the samples concern dating for archaeology, quaternary geology and marine geology. Major programs on Dissolved Inorganic Carbon (DIC) were started in both hydrology and oceanography. The largest hydrological project is a study of ground-water recharge in the semi-arid Kalahari region in Botswana. Only limited amounts of water were available dictating the use of AMS rather than our conventional laboratory [12].

For the ocean sciences, the Groningen Laboratory participates in the WOCE program. Vertical profiles of radiocarbon in dissolved inorganic carbon (DIC) were measured in the Southern Philippine Sea, during a cruise (PRIS)

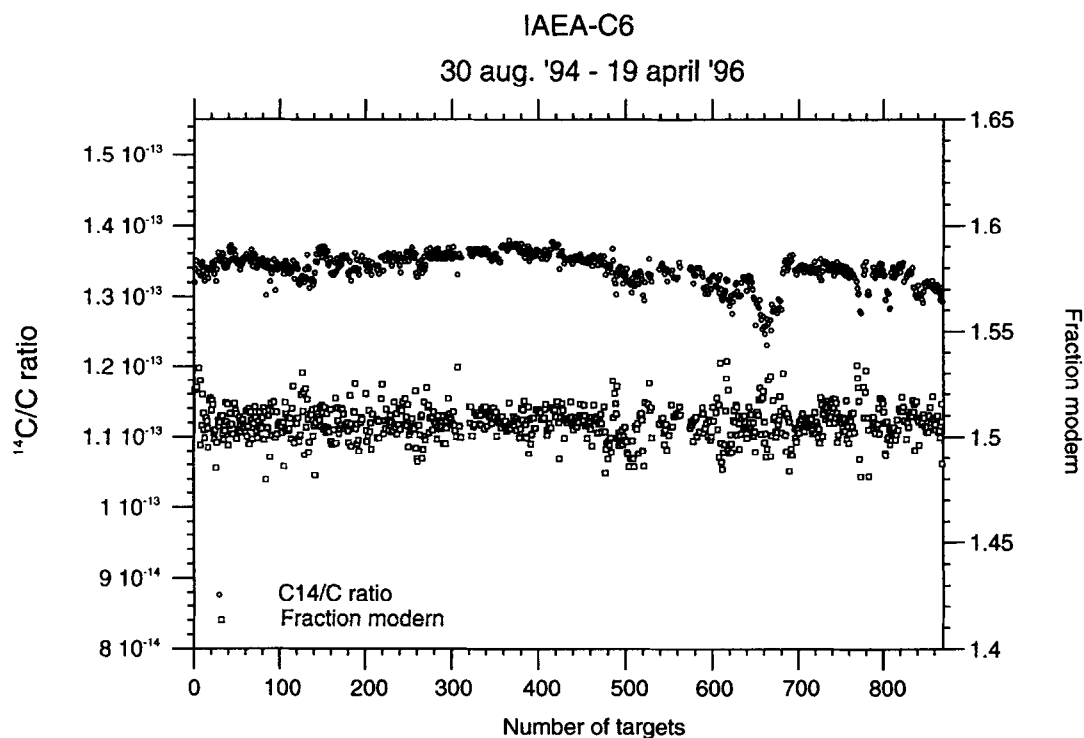


Fig. 3. ^{14}C measurements for about 900 IAEA-C6 (sucrose) standards, measured since acceptance of the AMS.

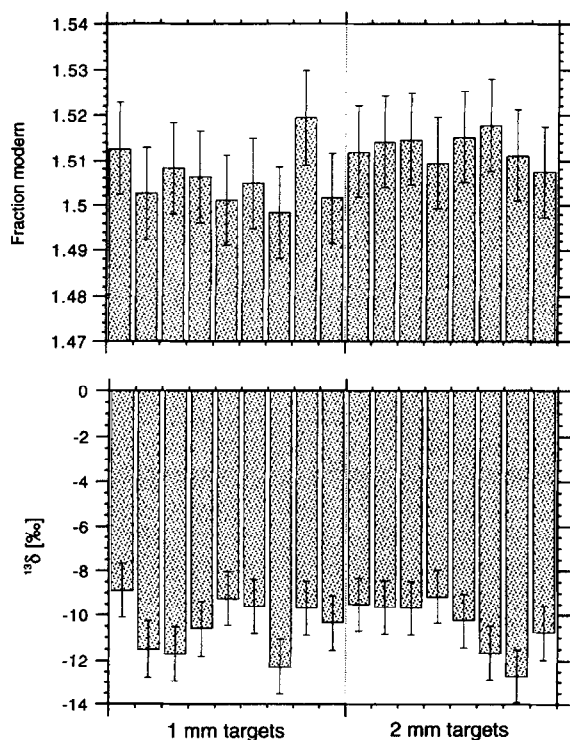


Fig. 4. Comparison between 1 and 2 mm targets C6 (sucrose).

from the Japan Marine Science and Technology Center. The CO_2 was extracted from the sea water in Japan, sealed in glass tubes and shipped to Groningen for ^{14}C analysis by AMS. A preliminary depth profile is shown in Fig. 5. The precision of the measurements was 5‰ or better.

Another major effort during the first two years of AMS operations (several hundred samples) was devoted to organic deposit layers from many sites: high-resolution data-

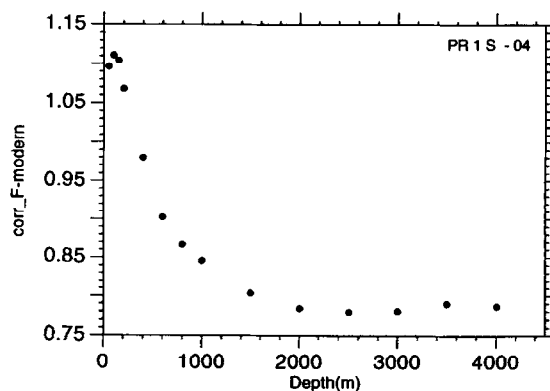


Fig. 5. Depth profile for ^{14}C -DIC from the Southern Philippine Sea.

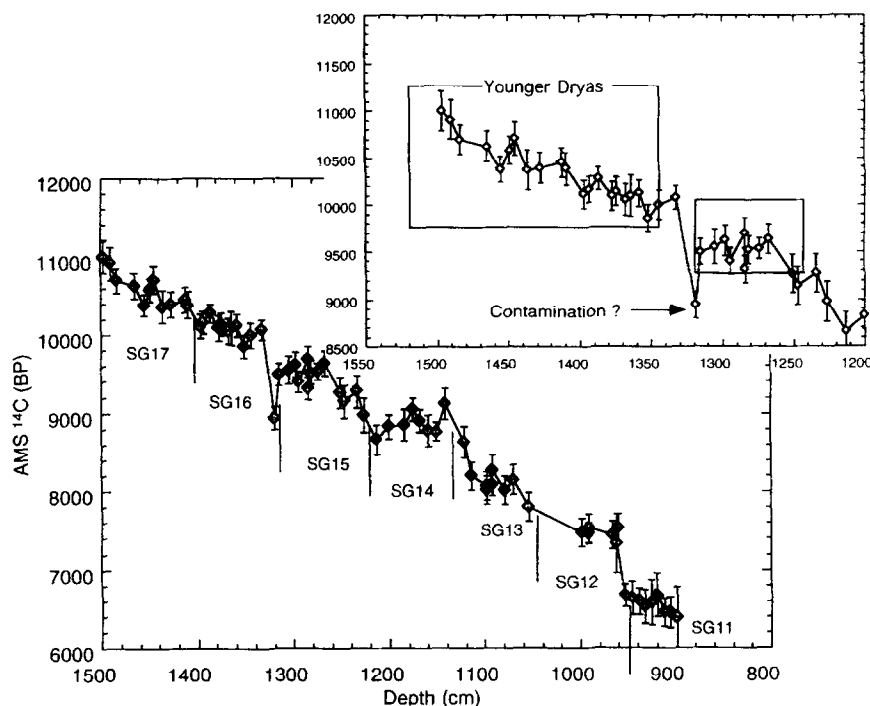


Fig. 6. AMS dates for terrestrial macrofossils from Lake Suigetsu in Japan. The insert shows the oldest part in detail, including the Younger Dryas.

ing of e.g. peat layers is made possible by AMS because of the small sample size, in combination with wiggle matching to the calibration curve. This technique was applied to several sites in NW Europe [13], concentrating on the “Hallstatt”-plateau (800–400 BC).

Laminated sediments can also only be dated by AMS, due to the small amount of organic carbon intrinsically present in these sediments. We started working on Lake Suigetsu from Japan [14]. Fig. 6 shows preliminary results: the ^{14}C dates as a function of core depth in cm. The Younger Dryas (YD) is tentatively indicated. One measurement (9000 BP, 1320 cm) is likely contaminated and indicated as such. The plateaux at 9600 and 10,000 BP are clearly recognized. A full analysis, combined varve counting and wiggle matching, is being conducted and will be published in the near future. Extension of this data set into the Late Glacial period will be measured shortly.

Three new programs were started, employing fractions which can only be dated by AMS. In the ocean sciences, we have started investigating Dissolved Organic Carbon (DOC), which analytically is very difficult to measure. For a report we refer to Ref. [15]. In addition we will study the origin and characterization of Particulate Organic Matter (POM) in estuaries [16] and in soil sciences, organic fractions based on size and density [17].

Finally, a substantial fraction of AMS time is devoted to atmospheric research. An “event trapping” experiment has been set up at a station near Groningen, where air is

sampled and trapped for off-line analysis of ^{13}C , ^{18}O and ^{14}C . This way, we were able to discriminate between biospheric and fossil fuel contributions of changes in the atmospheric CO_2 [18].

5. Conclusions and prospects

The Groningen AMS, dedicated to ^{14}C , is operational for two years and is presently running automatic batch measurements. Thus far we measured about 4000 targets; this number is limited by the target production capacity. The simultaneous analysis of all three carbon isotopes is an on-line diagnostic tool, which checks performance of the AMS continuously. We have shown that a precision below 0.5 pMC is obtainable on a routine basis. The background is routinely corresponding to 45 ka (0.2%) or better, and is mainly due to target fabrication; the machine background is negligible (> 100 ka).

Concerning the machine, the following modifications are planned. A weak point is the vacuum measurement at the high-energy side of the machine. We intend to replace the penning tubes by ion gauges. The stripper pressure performance will be improved by installing an electronic control unit, and replacing the O-rings by metal seals. A major effort for the time to come will be streamlining the data analysis package and AMS database setup. The Groningen

AMS will remain a dedicated ^{14}C machine for the foreseeable future; other isotopes are not planned.

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